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PATENT
CASE 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al : Group Art Unit: 1211
Serial No.: 08/360,184 : Examiner: E. White
Filed: December 20, 1994 :
For: Polyol Polyester Synthesis

DECLARATION UNDER 37 C.F.R. §1.608 OF EPHRAIM L. KELLY

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, EPHRAIM L. KELLY, declare that:

1. I have been employed by the assignee of the present application, The Procter & Gamble Company, since May 1967, and from March 1987 to the present, I have worked in the product development area.
2. From March 1987 to February 1990 I worked as a technician on the sucrose polyester synthesis project, and I was responsible for conducting sucrose polyester laboratory experiments wherein sucrose and fatty acid methyl ester were reacted to form sucrose fatty acid polyesters (sucrose polyesters) according to predetermined test plans and analyzing the results (or having the results analyzed under my direction and control) according to established procedures.

3. I conducted and analyzed the results of a variety of sucrose polyester laboratory experiments from March to October of 1989 under the direction and control of Mr. Patrick Corrigan and/or Mr. Richard Schafermeyer, including those described in this Declaration.

4. For each experiment which I conducted and which is described in this Declaration, I accurately recorded, on or about the day of the experiment, the general nature of the experiment, including pertinent reaction parameters, the results of the experiment, the date of the experiment and my signature in a Laboratory Notebook assigned to me for such a purpose.

5. I have examined Exhibit 35, and I confirm that Exhibit 35 comprises accurate copies of pages 54 and 57 to 59 of Laboratory Notebook SI 1373 on which I accurately recorded a series of experiments I conducted on March 15, 23 and 29 and April 4, 1989, and which I signed and accurately dated upon the completion of the procedures described on each page respectively. Laboratory Notebook SI 1373 was in my possession and control for at least the period of from October 1988 to November 1989.

6. Page 54 of Exhibit 35 describes a sucrose polyester reaction which I conducted on March 15, 1989 and which was part of a matrix of experiments intended to evaluate, inter alia, the effect of using residual potassium hydroxide (KOH) in the potassium soap emulsifier as the only catalyst in the reaction of sucrose and fatty acid methyl ester (i.e., potassium carbonate catalyst was not added to the reaction, as described at line 1 of page 54). Lines 26 to 31 of page 54 detail the percentages of the respective sucrose esters (e.g., monoester through octaester, represented as 1-8, respectively) in and the average degree of esterification based on mole

fractions, I-bar, of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 54, the sucrose polyester product comprised about 94.6% octaester and had an average degree of esterification of about 7.92 at 5.5 hours.

7. Page 57 of Exhibit 35 describes a sucrose polyester reaction which I conducted on March 23, 1989 and which was also part of the matrix of experiments intended to evaluate, inter alia, the effect of using residual potassium hydroxide (KOH) in the potassium soap emulsifier as the only catalyst in the reaction of sucrose and fatty acid methyl ester. Lines 26 to 32 of page 57 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 57, the sucrose polyester product comprised about 90.2% octaester and had an average degree of esterification of about 7.87 at 5.5 hours.

8. Pages 58 and 59 of Exhibit 35 describe a sucrose polyester reaction which I conducted on March 29 and April 5, 1989 and which was also part of the matrix of experiments intended to evaluate, inter alia, the effect of using residual potassium hydroxide (KOH) in the potassium soap emulsifier as the only catalyst in the reaction of sucrose and fatty acid methyl ester. Lines 17 to 30 of page 59 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 59, the sucrose polyester product comprised about 64.6% octaester and had an average degree of esterification of about 7.55 at 12.5 hours.

9. I have examined Exhibit 36, and I confirm that Exhibit 36 comprises accurate copies of pages 79 to 81 of Laboratory Notebook SI 1373 on which I accurately recorded a series of experiments I conducted on May 9-11, 1989, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

10. Page 79 of Exhibit 36 describes a sucrose polyester reaction experiment which I conducted on May 9, 1989 and which was part of a matrix of experiments intended to evaluate, inter alia, the effect of various soap levels on the sucrose polyester reaction. The reactants included about 25 grams of sucrose and about 11 grams of potassium stearate soap, together with potassium carbonate catalyst and fatty acid methyl esters, as described in lines 1 to 3 of page 79. Based upon a molecular weight of 342 for sucrose and a molecular weight of 322 for potassium stearate soap, the molar ratio of soap to polyol for this experiment was about 0.46:1. Lines 26 to 30 of page 79 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 79, the sucrose polyester product comprised about 98.8% octaester and had an average degree of esterification of about 7.96 at 3.75 hours.

11. Page 80 of Exhibit 36 describes a sucrose polyester reaction which I conducted on May 10, 1989 and which was also part of the matrix of experiments intended to evaluate, inter alia, the effect of various soap levels on the sucrose polyester reaction. The reactants included about 25 grams of sucrose and about 8 grams of potassium stearate soap, together with potassium carbonate catalyst and fatty acid methyl ester, as described in lines 1 to 3 of page 80. Based upon

the molecular weights set forth in paragraph 11, the molar ratio of soap to polyol for this experiment was about 0.34:1. Lines 25 to 29 of page 80 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 80, the sucrose polyester product comprised about 97.7% octaester and had an average degree of esterification of about 7.94 at 3.75 hours.

12. Page 81 of Exhibit 36 describes a sucrose polyester reaction experiment which I conducted on May 11, 1989 and which was also part of the matrix of experiments intended to evaluate, inter alia, the effect of various soap levels on the sucrose polyester reaction. The reactants included about 25 grams of sucrose and about 10 grams of potassium stearate soap, together with potassium carbonate catalyst and fatty acid methyl ester, as described in lines 1 to 3 of page 81. Based upon the molecular weights set forth in paragraph 11, the molar ratio of soap to polyol for this experiment was about 0.42:1. Lines 26 to 30 of page 81 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 81, the sucrose polyester product comprised about 97.0% octaester and had an average degree of esterification of about 7.93 at 3.75 hours.

13. I have examined Exhibit 37, and I confirm that Exhibit 37 comprises accurate copies of pages 86 and 87 of Laboratory Notebook SI 1373 on which I accurately recorded an experiment I conducted on May 23 and 24, 1989, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

14. The experiment described at pages 86 and 87 of Exhibit 37 investigated, inter alia, the effect of lower later stage reaction temperatures in a sucrose polyester reaction, particularly the effect on the formation of difatty ketones as described at line 1 of page 86. Pages 86 and 87 set forth the temperature and pressure of the reaction, measured at fifteen (15) minute intervals, beginning at 9:30 a.m. and indicate that at 12:00 p.m., the reaction temperature was reduced from 135°C to 100° C for the remainder of the reaction. Lines 17 to 30 of page 87 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours into the reaction. Page 87 also sets forth difatty ketone content, in ppm, in the respective products. As indicated on page 87, the final sucrose polyester product comprised about 94.0% octaester and had an I-bar of about 7.89 at 13 hours, and had a final difatty ketone content of from about 217 ppm to about 234 ppm.

15. I have examined Exhibit 38, and I confirm that Exhibit 38 comprises accurate copies of pages 147 to 154 of Laboratory Notebook SI 1373 on which I accurately recorded a series of experiments I conducted on September 29 and October 2-6 and 10, 1989, and which I signed and accurately dated upon the completion of the procedures described on each page, respectively.

16. Pages 147 and 148 of Exhibit 38 describe a sucrose polyester reaction experiment which I conducted on September 29 and October 2, 1989. The reaction was part of a matrix of experiments intended to evaluate, inter alia, the effect of various fatty acid methyl ester to

sucrose molar ratios. The reactants included about 25 grams of sucrose and about 172.8 grams of fatty acid methyl esters, together with potassium carbonate catalyst and potassium soap emulsifier, as described at lines 1 to 3 of page 147. Based upon a molecular weight of 342 for the sucrose and a molecular weight of 296 for the methyl esters, the molar ratio of total ester reactant to esterifiable sites of the sucrose was about 1:1. Lines 19 to 30 of page 148 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 148, the sucrose polyester product comprised about 74.3% octaester and had an average degree of esterification of about 7.65 at 10.5 hours.

17. Pages 149 and 152 of Exhibit 38 describe a sucrose polyester reaction which I conducted on October 3, 1989 and which was also part of the matrix of experiments intended to evaluate, inter alia, the effect of various fatty acid methyl ester to sucrose molar ratios. The reactants included about 25 grams of sucrose and about 202 grams of fatty acid methyl esters (about 172.8 grams added initially plus about 29.5 grams added at 12:15 p.m.), together with potassium carbonate catalyst and potassium soap, as described at lines 1 to 3 and 12 of page 149. Based upon the molecular weights set forth in paragraph 17, the molar ratio of total ester reactant to esterifiable sites of the sucrose was about 1.16:1. Lines 22 to 29 of page 152 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 152, the sucrose polyester product comprised about 93.2% octaester and had an average degree of esterification of about 7.86 at 7.5 hours.

18. Pages 150 and 152 of Exhibit 38 describe a sucrose polyester reaction which I conducted on October 4, 1989 and which was also part of the matrix of experiments intended to evaluate, inter alia, the effect of various fatty acid methyl ester to sucrose molar ratios. The reactants included about 25 grams of sucrose and about 175 grams of fatty acid methyl esters (about 172.8 grams added initially plus about 2.16 grams added at 2:35 p.m.), together with potassium carbonate catalyst and potassium soap, as described at lines 1 to 3 and 12 of page 150. Based upon the molecular weights set forth in paragraph 17, the molar ratio of total ester reactant to esterifiable sites of the sucrose was about 1:1. Lines 10 to 20 of page 152 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 152, the sucrose polyester product comprised about 78.8% octaester and had an average degree of esterification of about 7.69 at 10.5 hours.

19. Pages 151 and 154 of Exhibit 38 describe a sucrose polyester reaction which I conducted on October 6, 1989 and which also was part of the matrix of experiments intended to evaluate, inter alia, the effect of various fatty acid methyl ester to sucrose molar ratios. The reactants included about 25 grams of sucrose and about 177 grams of fatty acid methyl esters (about 172.8 grams added initially plus about 4.32 grams added at 11:35 a.m.), together with potassium carbonate catalyst and potassium soap, as described at lines 1 to 3 and 12 of page 151. Based upon the molecular weights set forth in paragraph 17, the molar ratio of total ester reactant to esterifiable sites of the sucrose was about 1:1. Lines 1 to 11 of page 154 detail the percentages

of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 154, the sucrose polyester product comprised about 77.9% octaester and had an average degree of esterification of about 7.65 at 10.5 hours.

20. Pages 153 and 154 of Exhibit 38 describe a sucrose polyester reaction which I conducted on October 10, 1989 and which also was part of the matrix of experiments intended to evaluate, inter alia, the effect of various fatty acid methyl ester to sucrose molar ratios. The reactants included about 25 grams of sucrose and about 178 grams of fatty acid methyl esters (about 172.8 grams added initially plus about 6.48 grams added at 9:55 a.m.), together with potassium carbonate catalyst and potassium soap, as described at lines 1 to 3 and 12 of page 153. Based upon the molecular weights set forth in paragraph 17, the molar ratio of total ester reactant to esterifiable sites of the sucrose was about 1:1. Lines 13 to 23 of page 154 detail the percentages of the respective sucrose esters in and the I-bar of the sucrose polyester product of this reaction as a function of time beginning at 1.5 hours. As indicated on page 154, the sucrose polyester product comprised about 80.4% octaester and had an average degree of esterification of about 7.67 at 10.5 hours.

21. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States

Code and that such willful false statements may jeopardize the validity of the present application or any patent issued thereon.

Respectfully submitted,

By Ephraim L. Kelly
Ephraim L. Kelly

Date: 3/26/99

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